

EFFECTS OF MATERIALS TESTING REQUIREMENTS AND PLANNING FOR IMPLEMENTATION OF INITIAL TEST PROGRAM FOR LIQUID SALT INTERMEDIATE LOOPS



September 2006

D. F. Wilson

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Materials Science and Technology Division

**EFFECTS OF MATERIALS TESTING REQUIREMENTS AND PLANNING FOR
IMPLEMENTATION OF INITIAL TEST PROGRAM FOR LIQUID
SALT INTERMEDIATE LOOPS**

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ACRONYMS

ASME	American Society for Mechanical Engineers
DOE	Department of Energy
IHX	Intermediate heat exchanger
MSRE	Molten Salt Reactor Experiment
NGNP	Next Generation Nuclear Plant
ORNL	Oak Ridge National Laboratory
R&D	Research and Development
TBD	To be determined

EXECUTIVE SUMMARY

Efficient and corrosion resistant heat exchangers are required to convey heat to the thermochemical cycle process fluids. Two candidate working fluids are being considered: helium and molten salts. Compatibility issues associated with helium are relatively better known and are being addressed in the Next Generation Nuclear Plant (NGNP) research programs. Molten salts, which provide for better heat transfer characteristics, lower pumping powers, and allows for the use of low pressure loops, present different and less well understood compatibility issues at temperatures of interest. While Hastelloy-N has been codified for use with fluoride salts at temperatures up to 750°C and for a minimum service life of 30 years, the use of molten salts at temperatures greater than 750°C introduces new performance requirements.

In order to determine the viability of molten salts as a heat transfer medium for an intermediate salt loop, the materials' compatibility and mechanical properties issues of possible structural materials must be addressed. Because of the lack of data for materials compatibility with molten fluoride, chloride, and fluoroborate salts and candidate alloys at the temperatures of interest, an extensive test program is needed. It is recommended that the proposed materials and variants of these be first evaluated in static tests at more than one temperature to determine their compatibility with molten salts. These salts must be carefully purified to avoid reactions of the structural materials of interest with impurities and techniques must be developed to perform realtime monitoring of the salt. Once a subset of these materials has been found, their mechanical properties at temperature, if unknown, must be determined. Those that possess needed compatibility and mechanical properties must be evaluated in flowing thermal-convection loops in order to determine mass transfer effects. These proposed tasks are separated in four areas: (1) salt chemistry measurement and control, (2) identification of monolithic material and clad-material candidates, (3) behavior of materials under flow conditions, and (4) metallurgical and mechanical issues. Also, activities associated with these tasks are defined.

1.0 INTRODUCTION

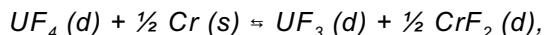
Efficient and corrosion resistant heat exchangers are required to convey heat to the thermochemical cycle process fluids. Two candidate working fluids are being considered: helium and molten salts. Helium poses relatively better known compatibility issues and requires higher pumping powers and pressures than molten salts. While the compatibility issues associated with high temperature helium are being addressed for the NGNP high temperature gas-cooled reactor, work in the current programs has not addressed issues associated with molten salts, which provide for better heat transfer characteristics, lower pumping powers, and allows for the use of low pressure loops. Previous programs established (specific code qualification) Hastelloy-N for use with fluoride salts at temperatures up to 750°C and for a minimum service life of 30 years. The use of molten salts at temperatures greater than 750°C introduces new performance requirements, which results in its own set of materials compatibility issues at these temperatures. The candidate salt coolants also possess relatively high (>350°C) melting points that must be accommodated by the system design. For example, LiFNaF-K melts at 454°C, KF-ZrF₄ melts at 390°C, and LiF-BeF₂ melts at 460°C.¹ In addition, two other classes of salts—alkali fluoroborate (represented by KF-KBF₄ 25-75 mol%), and alkali-halide (represented by LiCl-KCl, 59-41 mol%)—need evaluation. The latter two salts, along with LiF-NaF-KF, 46.5–11.5–42 mol%, represent the basic classes of salts (chloride, fluoroborate, and fluoride) that exhibit the necessary stability and differences in corrosion within each class of salt are not very distinct. In order to determine the viability of molten salts as a heat transfer medium for an intermediate salt loop, the materials' compatibility and mechanical properties issues of possible structural materials must be addressed. The viability program must::

- Identify candidate structural materials (alloys, composites, other advanced materials) for a molten salt intermediate loop at temperatures in the range of 750 to 950°C.
- Review available compatibility and mechanical property data to determine current limits of performance for candidate salts.
- Select the most promising structural materials/molten salt for the intermediate loop application.
- Develop a preliminary test matrix for evaluation of these materials.
- Perform the evaluation and down-select material(s).
- Perform long-term testing of down-selected material(s).

2.0 MECHANISMS OF CORROSION

Based on early research and development (R&D), the nickel-base alloy Hastelloy-N was developed by Oak Ridge National Laboratory (ORNL) for use in the Molten Salt Reactor Experiment (MSRE). Several hundred thousand hours of corrosion experience with Hastelloy-N and fluoride salts were obtained in thermal convection loop tests, \bar{z}^4 and pumped loop tests⁵ in addition to experience from the MSRE. Corrosion studies also included graphite as well as stainless steels and some refractory metal alloys, (e.g., TZM). While the understanding developed is based on fluoride salts, the principles have generally application to molten salts. Two general mechanisms of corrosion, metal dissolution and oxidation of metal to ions, can occur in molten salt systems. Because of low solubilities of most structural metals in salt systems, one mechanism, metal dissolution due to solubility in the melt, is not a common form of attack. The second mechanism, oxidation of metal to ions, is more likely. In addition to anodic dissolution and cathodic reduction of an oxidant, in the salts that are electronic as well as ionic conductors, reduction reactions can occur in the melt as well as at the metal-melt interface. In many molten salt systems the rate controlling step is ion diffusion from the alloy into the bulk solution, rather than charge transfer.

Thermodynamic stability of the salt components versus alloy constituents is quite important because molten-salt corrosion is usually induced by reduction/oxidation (redox) reactions. In work leading to operation of the MSRE, ORNL demonstrated the excellent compatibility of Hastelloy-N with fluoride salts containing LiF, BeF₂, ThF₄, and UF₄. If the salt is pure and the metal clean, UF₄ is the strongest oxidant in a fuel-salt system. The reaction,



has a favorable free energy of formation at 500 to 800°C. Since CrF₂ is one of the most stable structural metal fluorides, Cr can selectively react with extraneous (impurity) oxidants (fluorides) in the system. Because of the unusually high stability of alkali metal and beryllium fluorides, the corrosion potential of melts without uranium is likely controlled by redox equilibria involving impurities in the melt or gas phase, or a soluble redox buffer (M^{3+/2+}) that is specifically added to the system. Gradients in the chemical activities of constituents caused by temperature differences can result in dissolution of metal in one region of the system with subsequent deposition in other portions of the

system. The amount of attack will depend on the driving force and dissolution or deposition kinetics for reactions that result in transporting the corroding species to a different part of the loop circuit where they might deposit. In single material systems (e.g., capsule tests) at constant temperature, there would be no activity gradient and the amount of attack would be a function of the relative solubility of the respective corrosion product in the salt or, if the product is volatile, the partial pressure in the atmosphere immediately above the salt. Thus, equilibrium solubility principles would limit the amount of solute and degree of corrosive attack in this type of system. However, a non-isothermal system could also be subject to corrosion from thermal gradient mass transfer if the chemical potential of the corrosion product fluoride, at a given concentration, is a strong function of temperature, and deposition of the corrosion species occurs in the cooler regions of a loop operating with a relatively high temperature differential. Although thermodynamics limits the amount of solute in a specific volume of salt in a closed system (capsule test) at constant temperature, continued transport of material can occur to the cold region of a non-isothermal system.

Mass transfer can also occur when dissimilar structural materials are included in the same system. The dissimilar materials do not have to be electrically coupled. Two things are required for dissimilar material mass transfer to be a factor. First, an element contained in one of the materials has to have a strong tendency to form an alloy or compound with the second material. More importantly, an element in one of the materials must be subject to oxidative attack or suffer dissolution within the salt solution. The oxidative attack can be either by reaction with impurities or, if a reactive element, with the salt constituents. In either event, once the element goes into solution, it can then migrate through the solution and form the required product if the chemical driving force (activity gradient) is sufficient. If the product does form, this will allow the mechanisms of corrosive attack or dissolution to continue (i.e., favorable thermodynamics) although kinetics may be limited by solid state diffusion. If the attack is solely by reaction with impurities, the problem may dramatically slow with time. However, the reaction will not stop if there is solubility in the salt for a specific element.

Even if redox reactions with major salt constituents are limited, materials can interact with oxidizing impurities present in the salt or in the system (capsule or loop). Moisture or oxides residing on the container or loop walls, can all lead to adverse corrosive

reactions. In order to control the redox potential of the fluoride salt in the MSRE, the melt chemistry was made slightly reducing by the addition of a small amount of beryllium metal that reduced approximately 1% of the UF_4 solute to UF_3 :



As a result of this addition, the 100/1 ratio of UF_4/UF_3 minimized the corrosion of Hastelloy-N by UF_4 (where Cr is the most active metal present and therefore the first to be oxidized) because of the equilibrium between the chromium and uranium components:



The redox potential of systems that do not contain uranium may be controlled in another fashion – by addition of a small amount of solute that can exist in two oxidation states, by addition of a sparge gas that is set at the proper redox potential, or by electrochemically setting an applied potential equivalent to the desired redox potential. These approaches need to be evaluated.

3.0 CANDIDATE MATERIALS FOR USE WITH MOLTEN SALTS

While a candidate list for use with molten chlorides and fluoroborates cannot be presented at this time, based on past experience, an initial candidate list of materials for evaluation in the chosen fluoride salt is proposed. This list is presented in Table 1. Alloys high in chromium content may only be viable in very low impurity content salt that is maintained at the proper redox (reducing) state. An extensive literature evaluation needs to be performed to assist in the development of a similar list for chloride and fluoroborate salts.

Table 1. Potential candidate materials for use in molten fluoride salts

Material	Fluoride salt corrosion resistance	Fabricability	Highest use temperature °C	Comment
Haynes 242	Very good	Good	540	
Hastelloy-N	Excellent	Good	800	
Hastelloy X or XR	Needs evaluation	Good	900	May need a protective coat
Inconel 617	Needs evaluation	Good	900	ASME Sect. VIII Div. 1 May need a protective coat
Haynes 230	Fair	Fair	899°C	ASME Sect. VIII Div. 1 May need a protective coat
Haynes 214	Very good	Poor-fair	1000	
MA 956	Good	Poor-fair	1300°C	No longer in production. May need a protective coat
MA 754	Very good	Poor-fair	1177°C	No longer in production
Hastelloy B3	Needs evaluation	?	?	Service temperature limited by brittle intermetallic formation. Use below 750°C.
Cast Ni superalloys	Very good		>900°C	For pumps. May need protective coat
Nb-1Zr	Very good		>900°C	
SiC (CVD)	Very good	poor	Above that needed	Need a near net shape and the ability to seal

4.0 TESTING

Because of the lack of data for materials compatibility with molten fluoride, chloride, and fluoroborate salts and candidate alloys at the temperatures of interest, an extensive test program is needed. It is recommended that the proposed materials and variants of these be first evaluated in static tests at more than one temperature to determine their compatibility with molten salts. These salts must be carefully purified to avoid reactions of the structural materials of interest with impurities and techniques must be developed to perform realtime monitoring of the salt. Once a subset of these materials has been found, their mechanical properties at temperature, if unknown, must be determined. Those that possess needed compatibility and mechanical properties must be evaluated in flowing thermal-convection loops in order to determine mass transfer effects. These proposed tasks are separated in four areas.

4.1 Salt Chemistry Measurement and Control

Thermodynamic stability of the salt components versus alloy constituents is quite important because molten-salt corrosion is usually induced by reduction/oxidation (redox) reactions. As a result, an alloying element will tend to go into solution if it forms a more stable species than those that exit in the melt. Hence, impurities present in the salt, or the gas phase, as well as moisture or oxides residing on the containment walls, can all lead to adverse corrosive reactions. Thus it is necessary that the concentration and identity of impurities in the melt be well characterized so that:

- Adverse reactions from impurities do not cloud an understanding of the materials salt interaction.
- Electrochemical and spectro-chemical instrumentation can be further refined to allow for real-time monitoring of corrosion effects in an operating system and to provide an understanding of the corrosion mechanism (controlling factors).
- On-line, side-stream clean-up of salts in an operating reactor can be performed efficiently and effectively.
- Techniques be implemented that may allow for control of redox potential of the salt and hence the corrosion rate of structural materials.

4.2 Identification of Monolithic Material and Clad-material Candidates

There is no well characterized material for operation above 750°C. Materials are

required especially for pumps, because a “low wear” contact surface material would be required. There are needs for:

- Cast high-nickel material with/without a hard surface coating.
- Coatings (cermets, carbides) for pump bearings immersed in salt and valve seats.
- Graphite, and C/C and SiC/SiC composites for use in the presence of alloys in the salt.

Corrosion effects must be evaluated so that:

- Identity and concentration of alloy element migrating into the salt can be ascertained (thermodynamic stability effects).
- Effect of dissimilar materials (activity gradients) in the salt on the corrosion of each other can be ascertained.
- Identity of diffusing species from a high strength substrate through a corrosion resistant cladding can be ascertained and efficacy of possible diffusion barriers determined.
- Down selection of materials of appropriate strength and identification of alloying elements that must be controlled can be performed.

4.3 Behavior of Materials under Flow Conditions

Because an operating system is not isothermal, corrosion reactions driven by thermodynamic considerations or impurity reactions, which can have different solubility limits at various temperatures, can have additional impacts. Alloying elements, which can go into solution at one temperature and come out of solution at a different temperature, can build up in critical flow paths and created significant porosity at other sites. This is particularly applicable to piping and heat exchangers. Hence evaluation of temperature gradient assisted corrosion effects must be initiated.

4.4 Proposed Test Matrix

A proposed materials test matrix is presented in Table 2

Table 2. Static capsule tests for each material of choice in each salt of choice for down selection of materials and thermal convection loop tests for down-selected materials

Test	Temperature (°C)	Time (h)	Comments
Mono-material static capsule	750	100	Screening tests. Allow for evaluation of impurities in molten salt versus thermodynamic stability
	750	500	
	850	100	
	850	500	
	950	100	
	950	500	
	1000	100	
	1000	500	
Dissimilar materials static capsule Material A for capsule and materials B for specimen (much smaller surface area)	750	500	Determine thermodynamic chemical activity driven corrosion effects. (Assumes that dissimilar materials will be employed in the molten salt environment.) Use materials that pass above screening. Repeat if more combinations of materials will be exposed to the molten salt environment
	850	500	
	950	500	
	1000	500	
	750	500	
	850	500	
	950	500	
	1000	500	
Down-selected mono-material thermal-convection loop	Gradient from 1000 to 725	3000	Determine temperature driven mass transfer. Electrochemical monitoring of salt reservoir during test; periodic chemical analyses of salt content; post-test evaluation of specimens and loop Repeat for each down-selected material

Table 2. Static capsule tests for each material of choice in each salt of choice for down selection of materials and thermal convection loop tests for down-selected materials

Test	Temperature (°C)	Time (h)	Comments
Final down-selected mono-material and bi-materials thermal-convection loop			
Down-selected bi-materials thermo-convection loop	Gradient from 1000 to 725	10,000	Loop made of one material. Specimens made of a different material. (Assume that different materials will be employed in the molten salt.) Determine additional effects of thermodynamic activity driven mass transfer.
Final down-selected bi-materials thermal-convection loop	Gradient from 1000 to 725		Electrochemical monitoring of salt reservoir during test; periodic chemical analyses of salt content; post-test evaluation of specimens and loop Repeat for each down-selected combination of materials chosen

4.5 Metallurgical and Mechanical Issues

In addition to the major compatibility issues, the candidate structural materials are expected to be exposed to temperatures up to 950°C under certain loading conditions. Therefore, common issues for high temperature applications must also be considered. The consideration should include the highest service temperature and strength, aging effects on microstructural stability and mechanical properties, effects of product form and grain size, manufacturability, and American Society of Mechanical Engineers (ASME) codification. If the candidate material is already considered for metallic reactor internal or helium-helium IHX, most of the metallurgical and mechanical property issues should be complementary. Therefore, only limited additional information will be required for the molten salt IHX application, which may include optimum metallurgical conditions for improved performance specific to molten salt IHX design, microstructural stability and mechanical properties degradation in molten salt environment. For candidate materials

that are not considered for use in the helium environment, all the issues related to high temperature services must be addressed. To efficiently study the metallurgical and mechanical fitness for molten salt IHX application, the investigation and testing should be closely coordinated with activities addressing corrosion and compatibility to provide adequate information for eliminating unqualified materials at the earliest stage.

4.6 Proposed R&D to Address Metallurgical and Mechanical Property Issues

As the first step, a comprehensive and detailed review of selected candidate materials that are not covered by activities within the helium environment must be conducted. The existing database for those materials will be assembled, analyzed and evaluated with respect to the design and operating requirements for molten salt IHX. Principal topics for review will include high temperature strength, metallurgical stability, long-term performance under molten salt condition, effects of molten salt on the mechanical and physical properties, fabrication, codification status and needs, including maturity and limitations of the high-temperature design methodology for each candidate material. The review will provide guidance for further defining R&D focuses. At present, the activities are proposed as listed in Table 3. These activities may be refined when more information becomes available.

4.7 Activities

Proposed activities are presented in Table 3.

Table 3. Proposed activities for molten salt IHX application

Year	Description	Comments
Salt chemistry measurement and control		
FY07	Evaluate electrochemical approaches to salt chemistry analysis	Select and evaluate various electrochemical probes (with both reference and quasi-reference electrodes) for use in molten salt test systems. Measure and report electrochemical signatures of key corrosion species in salt solution.
FY07	Perform literature evaluation of material compatibility issues, salt purification approaches, and redox -control for molten chloride and fluoroborate salts	Determine feasibility of use of chloride and fluoroborate salts
FY08	Continue evaluation of electrochemical techniques	
	Evaluate spectrochemical technique for identification of corrosion species in molten salt	Establish spectrochemical measurement apparatus for identification of key corrosion species in molten salt solution. Conduct measurements which show the effect of different salt environments (Lewis acidity, redox state of salt).
FY09 and beyond	Continuation of electrochemical and spectrochemical work to be determined (TBD)	
Identification of monolithic material and clad-material candidates		
FY07	Evaluate performance potential materials at various temperatures (capsule tests) for fluoride salt	Mono-material evaluation
FY08	Evaluate performance potential materials at various temperatures (capsule tests) for fluoride	Down selected materials in dissimilar material evaluation testing
FY08	Evaluate performance potential materials at various temperatures (capsule tests) for chloride or fluoroborate salt	Mono-material evaluation
FY09	Evaluate performance potential materials at various temperatures (capsule tests) for chloride or fluoroborate salt	Down selected materials in dissimilar material evaluation testing
FY09 and beyond	Continuation of down-selection work TBD	

Table 3. Proposed activities for molten salt IHX application

Year	Description	Comments
Behavior of materials under flow conditions		
FY07		Activities are depended on results of static capsule test
FY08	Design and build system, and initiate testing under flow conditions	Mono-metallic loop
FY09 and beyond	Continuation of mono-material and start of dissimilar material testing TDB	
Metallurgical and mechanical issues		
FY07	Conduct a comprehensive review of metallurgical and mechanical issues for candidate materials for molten salt IHX application	Corrosion screening of the candidate materials will be on-going in FY07.
FY08	Acquire and age the materials down-selected by corrosion screening in FY07	Depending on corrosion screening in FY07, environmental aging may also be considered, which will drive up the cost.
	Investigate weldability of materials down-selected by corrosion screening in FY07	It is anticipated that most of the candidate materials will be difficult to weld and would require significant effort.
FY09 and beyond	Work on welding, mechanical testing, microstructural evaluation of materials TBD	

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